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2 February 2004

Dear Sir,

SUBMISSION OF PRIORITY DOCUMENTS

Serial Number: 10/694,835

Confirmation Number: 2479

Art Unit: 1744

Applicant: DARLINGTON, Alan Blake et al.

Our File Ref: 221-46US

We now submit the certified copy of the priority document in respect of the above patent application.

Submitted by,

Anthony Asquith
(Regn 32373)
Agent for the Applicant

Enclo:

Certified Copy (GB-0225230.2)



INVESTOR IN PEOPLE

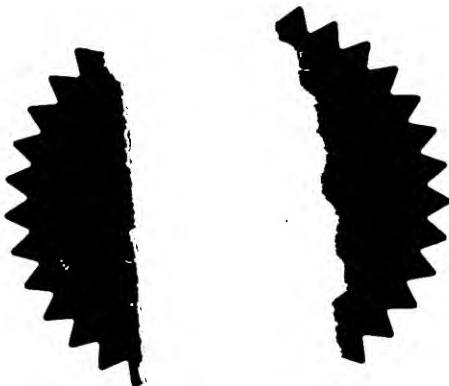
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In accordance with the Patents (Companies Re-registration) Rules 1982, if a company named in this certificate and any accompanying documents has re-registered under the Companies Act 1980 with the same name as that with which it was registered immediately before re-registration save for the substitution as, or inclusion as, the last part of the name of the words "public limited company" or their equivalents in Welsh, references to the name of the company in this certificate and any accompanying documents shall be treated as references to the name with which it is so re-registered.

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(Rule 6)



300CT02 E759674-1 D02986
P01/7700 0.00-0225230.2

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Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

1. Your reference

221-45GB

30 OCT 2002

2. Patent application number

(The Patent Office will fill in this part)

0225230.2

3. Full name, address and postcode of the or of each applicant (underline all surnames)

University of Guelph
Guelph
Ontario N1G 2W1

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

798256001

4. Title of the invention

PLANT BASE SYSTEM FOR
ABATEMENT OF GASEOUS AMMONIA CONTAMINATION

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent (including the postcode)

Anthony Aswith
328 Leeds Rd
Scholes
Leeds LS15 4DD

Patents ADP number (if you know it)

6562201003

6. If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number

Country

Priority application number
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Date of filing
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Number of earlier application

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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:

- a) any applicant named in part 3 is not an inventor, or
- b) there is an inventor who is not named as an applicant, or
- c) any named applicant is a corporate body.

See note (d))

1
INVENTION DISCLOSURE

PLEASE COMPLETE ACCURATELY AND FULLY. THIS INFORMATION IS NEEDED FOR AN ACCURATE AND TIMELY EVALUATION OF YOUR INVENTION. IF ADDITIONAL SPACE IS NEEDED ATTACH EXTRA PAGES.

1. NAME OF INVENTION: Plant based system for abatement of gaseous ammonia contamination.

2. INVENTORS: Give the names and positions of all individuals (faculty, research associates, post doctoral fellows, staff, students, etc.) who may have made an intellectual input into the invention. Those who simply followed instructions would not qualify as inventors. If there is any doubt include the name and mark with a "?".

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3. BRIEF DESCRIPTION: Attach a brief but thorough description of your invention. Attach supporting information that may help to explain the ideas, such as plans, sketches, photographs, drawings, flow sheets, performance data or graphs.

Gaseous ammonia is a common by product of industrial and agricultural processes. Sources include the production of industrial chemicals, fertilizers, refrigerants, synthetic fibres, explosives, food additives and cleaning agents. It is a common pollutant.

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in indoor air and in the environment at large. Atmospheric release of ammonia in Canada is estimated to be 18 000 tonnes per year. Ammonia gas is a precursor to photochemical smog and is generally regarded as a nuisance gas when released into the environment. Environmental discharge guidelines have been set as $3600 \mu\text{g m}^{-3}$ (half hour average) at the point of impingement. Ammonia concentrations can be quite high due to industrial and agricultural processes, $53\ 000 \mu\text{g m}^{-3}$ were measured in a fertilizer plant and $35\ 000 \mu\text{g m}^{-3}$ in a swine facility. Pigs exposed to elevated ammonia levels are less able to gain weight (Drummon 1980). Elevated concentrations produce headaches, nausea and loss of appetite in humans and occupational guidelines limit exposure to no more than $100 \mu\text{g m}^{-3}$ in a 24 hour period.

The current occupational and environmental guidelines limit the amount of ammonia in terms of exposure to employees and discharge into the environment. Exposure to building occupants is typically controlled through increased ventilation. This approach can become energy intensive, particularly where the outdoor conditions requires that "fresh air" be treated in terms of temperature and humidity to maintain occupant comfort. Traditional biofiltration has been employed as a form of emission reduction. However, these are subject to pH changes which limit the operating life and efficiency of these systems. These systems take advantage of the solubility of ammonia in water by bubbling ammonia contaminated gas through water traps. Subsequent treatment of the waste water is required in these applications.

This disclosure is the product of meetings between Alan Darlington and Stefan Richard as they were establishing research objectives for a graduate degree in September 2001. The proposed biofiltration system is designed to remove gaseous ammonia from exhaust or re-circulating air streams. It features a community of plants and mosses grown either vertically or horizontally on a porous medium. The media may be a mixture of synthetic or natural fibres or any combination which provides low resistance to air flow. Air flow rates can be varied to suit particular applications by adjusting the re-circulating or exhaust fan used to pull air through the system. As contaminated air passes through the media, ammonia is absorbed from the air stream into the aqueous phase attached to the media.

As discussed above, the idea of using biofilters to remove gaseous ammonia is not new, however, incorporating plants into biofilters for ammonia removal is original. In theory the incorporation of plants in the system will act as a sink for nitrogen. While existing systems have used microbial communities to eliminate ammonia from waste gas streams, those systems are prone to acidification. This is typically a result of the microbial transformation of NH_3 to HNO_3 . The inclusion of higher plants in this system acts as an alternate nitrogen sink in the system and prevents pH changes. Ammonia is chemically broken down through a variety of microbial and botanical metabolic processes and eventually assimilated into the biomass of the system. Plants have a biological need for nitrogen, and will assimilate from the environment as part of their normal metabolic process. Since ammonia is transformed into benign forms of nitrogen, and can be safely disposed of as excess biomass, unlike water traps where ammonia is simply stored and must be treated. The improvements from that arise from the inclusion

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of plants are, increased ammonia removal from waste gas streams, biofilter stability and increased operational life and the creation of only organic waste products.

4. DESCRIPTION ANALYSIS: Review the description in section 3 and ensure the following questions are clearly answered - if not, give the additional information below or on extra pages.

4.1 What problem does this invention deal with or help solve?

Elimination of gaseous NH_3 from contaminated air streams.

4.2 By what means has this problem been dealt with up to the present?

Indoor ammonia levels are controlled through ventilation. Atmospheric discharge of ammonia is controlled through conventional, non plant based biofiltration or through water traps.

4.3 What are the limitations or drawbacks of present methods or products?

Current solutions may be costly in terms of temperature and humidity conditioning in buildings or agricultural facilities. The employment of traditional biofilters are subject to pH changes which limit their operational lifetime and ammonia removal efficiencies, furthermore the inclusion of higher plants in biofiltration systems adds a nitrogen sink which improves the overall performance of the system. Water traps require further treatment to eliminate ammonia.

4.4 What capabilities of your invention overcome such limitations? and how?

When employed in a recirculating fashion the system can be used to improve air quality in buildings or facilities without incurring the costs of additional ventilation. The inclusion of plants in the system provides an additional sink for nitrogen compounds enhancing traditional biofilter technologies. The result is a more stable pH leading to a long biofilter life and greater ammonia elimination before discharge or recirculation. This technology does not require further treatment processes to eliminate ammonia, all waste products can be disposed of safely in landfills or compost facilities.

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4.5 Identify particular aspects of your invention that you believe to be original.

The use of biofilters to treat ammonia vapor is not original. However, the inclusion of plants into the system is new. As stated above the inclusion of plants into biofiltration systems is known to improve their efficiency, in this case the biological demand for nitrogen compounds by the higher plants will greatly improve the efficiency and lifetime of biofilters.

4.6 What attractive features does your invention offer the user?

Cost reduction in terms of ammonia treatment, and ventilation costs. Air quality improvements for employees or livestock. Aesthetically pleasing.

5. USES AND APPLICATIONS:

5.1 What are the immediate uses for the invention and by whom?

There are several applications of this invention. If applied in a recirculating fashion the system could be used in any manufacturing facility, workplace, mine or agricultural facility where gaseous ammonia is released. In all of these cases the invention would be used as an indoor air quality tool, designed to improve the quality of indoor air by removing ammonia vapours. The direct cost savings to the user would come in the form of reduced ventilation costs and increased worker productivity. In agricultural applications improved air quality will reduce ventilation costs and may lead to greater weight yields in livestock. The system could also be applied as an ammonia scrubber before discharge to the environment. This technology would aid manufacturing or chemical plants which produce ammonia in industrial applications to meet emission guidelines.

5.2 Describe briefly future applications you foresee if there are any.

Advanced life support and residential applications.

6. DEVELOPMENT STATUS:

5.

9. MARKET INFORMATION:

9.1 CURRENT COMPETITION: What existing products or processes do the same job and why is yours better?

Conventional air handling systems rely on ventilation with 'fresh' outdoor air to maintain indoor concentrations of ammonia. Importing outdoor air represents a major operational cost for most buildings, since, imported air must be conditioned in terms of temperature and humidity to maintain occupant comfort. Modern buildings, in general have been designed to minimize air exchanges to reduce these costs. When operated in a recirculating fashion, this system can reduce the need for ventilation, without requiring additional conditioning. When exhausted to the environment ammonia is a nuisance gas, creating odor problems and contributing to photochemical smog. Application of the biofilter in the exhaust stream can be used to reduce these emission and thus environmental impacts of ammonia. The conventional approach to ammonia abatement relies on physical/chemical scrubbing, which ultimately produces toxic wastes that must be disposed of.

9.2 PROJECTED MARKET: Who are the prospective users of your invention?

a) Major users:

- 1) Hog, cattle and poultry facilities
- 2) Mushroom farms
- 3) Industrial and commercial buildings
- 4) Mines where ammonia gas is produced through the use of explosives

b) Secondary users:

- 1) other agricultural applications
- 2) small animal facilities
- 3) controlled environment or life support systems.

6.

AMMONIA BIOFILTRATION OF AIR IN CLOSED SYSTEMS

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ABSTRACT

There is an increasing public awareness surrounding issues of air quality in occupied settings. Environmental and financial concerns with respect to energy inputs have forced designers and engineers to construct buildings that are ever more sealed from the outdoors. However, this allows for accumulation of airborne pollutants such as ammonia in residential and commercial settings. The most common sources of ammonia are biological processes that break down organic waste and cleaning solutions that contain ammonia. Any natural or industrial process that concentrates nitrogen-containing organic matter and makes it available for decomposition becomes a potential source for high ammonia production. The accumulations of ammonia in closed settings are of concern in the agricultural industry, the mining industry and in advanced life support systems where ventilation as a means to maintain air quality is not always feasible. Biofiltration (biological degradation of pollutants by passing a polluted airstream through a bed of beneficial microbes) of air in occupied settings has been proposed as an alternative to ventilation. In this study, "bench top" biofilters are exposed to a range of ammonia concentrations (0.5 to 50 ppmv) and the ability of the biofilter to remove ammonia from a closed system is assessed. The biofilters used in this study are hybridizations of phytoremediation (the biological degradation and accumulation of pollutants by plants) and traditional microbial biofiltration technologies.

INTRODUCTION - INDOOR AIR QUALITY

Studies by the Canadian Department of the Environment indicate there are 5000 deaths each year across Canada due to air pollution (ENVCAN, 2001). The Ontario Medical Association states that air pollution cost to the taxpayers of Ontario is more than \$1 billion annually in hospital admissions, emergency room visits and absenteeism (ENVCAN, 2001). In 1999, Canada passed the Canadian Environmental Protection Act (CEPA). This act has, among other things, set aside \$2.65 billion to fund new infrastructures focused on problems surrounding clean air, water and wastewater.

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Air pollution also plays an important role in all indoor settings. Since Americans spend upwards of 90% of their time indoors (USEPA, 1989), indoor air quality is of great importance. One such important indoor pollutant is ammonia (NH_3). Ammonia is a colourless gas that has a pungent odour when in concentrations of 0.043 to 53 ppm. Short-term exposure to low levels of inhaled ammonia (0 to 27 minutes at 57 to 500ppm) can cause nasal and pharyngeal irritation, but no tracheal irritation (OMoFE, 2001). Short-term exposure to high levels of inhaled ammonia (acute exposure) can cause irritation to the eyes, skin, nose, mouth, throat or the lungs. Chronic Exposure to low levels of inhaled ammonia (40ppm) has been reported to result in headaches, nausea and reduced appetite (USEPA, 1989). Exposures to ammonia levels of 2.5 parts per thousand (ppt) or above can be life threatening (OMoFE, 2001).

Ammonia is one of the most extensively used industrial chemicals in North America (WHO, 1990). The 1990 inventory, Ammonia Emissions in Canada, found that atmospheric ammonia emissions were 651 kilo tonnes, 87% from agriculture of which 82% from animals and 18% from fertilizer application (Chambers, 2001). Between 1993 and 1997, the *National Pollutant Release in Canada* reports ammonia atmospheric releases to average 18000 tonnes per year in Canada (OMoFE, 2001). Between 1993 and 1997, ammonia was one of five highest emitted substances in Ontario (OMoFE, 2001).

Ammonia in Closed Systems

Accumulation of ammonia is of concern for several indoor environments (Closed Systems) including agriculture systems, advanced life support systems, mining industry and residential dwellings.

Agricultural Settings - Indoor agricultural settings such as livestock barns, swine facilities, poultry houses and mushroom farms often are found to contain NH_3 at levels above those considered to be safe for human exposure (Ni, J.-Q., 2000). Risk to humans is not the only factor relevant when considering NH_3 levels in indoor agricultural settings. At 50ppm, a 12% weight-gain reduction was observed in swine (Drummond, 1980). Although no detrimental health effects were observed in the swine, this loss in weight-gain suggests a loss of productivity for the growing facility.

Advanced Life Support Systems - Advanced life support systems presents a unique situation where exchange of contaminate air with "fresh" air is not always possible or feasible. NASA has established criteria for spacecraft maximum allowable concentrations for human health (SMAC) for air pollutants. SMAC values for NH_3 are 30 and 20 ppm for 1 hour and 24 hours respectively, and the value is 10 ppm for 1 week to 6 months (Perry, 1998). The European Space Agency's Micro-Ecological Life Support System Alternative (MELiSSA) project examines the use of complex ecosystems as tools that can provide life support functions for a crew of astronauts. The main functions of MELiSSA would be recycling waste (inedible biomass, faeces and urine), carbon dioxide and minerals, and also to produce food and air revitalization (including the removal of ammonia from contaminated airstreams).

Mining Industry - Ammonia accumulation can be problematic in active mine shafts where blasting has occurred. Ammonia is produced as a by-product of blasting with ammonium nitrate. Contaminated air must then be pumped out of the shaft while fresh air is pumped in. This process requires much energy and can be very costly.

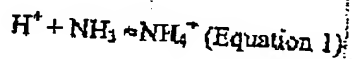
Residential Dwellings - Since the early 1990's, homes built in Finland have been tested for NH_3 . This testing was initiated due to symptoms expressed by the residents. Results of testing 632 dwellings indicate that 38.3% of the homes contained NH_3 levels that exceeded $40\mu\text{g}/\text{m}^3$ (approximately the lower limit of odour threshold). Concentrations of NH_3 above this level have been deemed to pose risk to humans by the Finnish Ministry of Social Affairs and Health (Hiltunen, K. 2001).

To avoid the accumulation of indoor air contaminants (including ammonia), much energy and cost are expended in providing adequate IAQ (indoor air quality). Standard treatments for maintaining adequate IAQ involve frequent air exchange with a building's external environments (ventilation). This new air must be conditioned in terms of its temperature and humidity prior to distribution within the space, requiring substantial energy input. With increasing energy cost, new technologies should be considered to treat indoor air. Biological treatments of the indoor air are emerging as one such new technology.

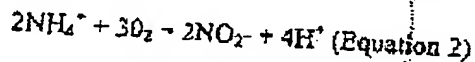
Degradation of Ammonia (Nitrification)

Nitrification is a two-step process that results in the reduction of nitrogen from an oxidation state of -3 to an oxidation state of +5. NH_3 is naturally transformed in soils and water by autotrophic nitrifying bacteria. This bacterium uses NH_3 as an electron source, CO_2 as a carbon source and O_2 as an electron acceptor.

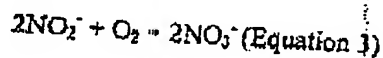
Since the systems that will be examined will contain water, it is important to consider not only ammonia, but also ammonium (NH_4^+).



The first step of nitrification is the transformation of $\text{NH}_3/\text{NH}_4^+$ to NO_2^- by an ammonia/ammonium oxidizing bacteria such as *Nitrosomonas*.



The second step is the transformation of NO_2^- to NO_3^- by *Nitrobacteria*.



Biofiltration of Ammonia

Ammonia can be successfully degraded/reduced using biofilter technologies for conventional "end of the pipe" treatment systems. However, very little work has been done on biofiltration of ammonia in indoor or closed settings where the air streams are continually recycled. Joshi et al., 2000 used biofilters containing perlite inoculated with nitrifying enrichment culture to treat airstreams in closed systems that were contaminated

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with ammonia at levels of 20 to 50 ppm. These biofilters removed 99.99% of the ammonia in the contaminated air streams over 101 days.

In this study, "bench top" biofilters were exposed to ammonia contaminated air streams (50ppm v/v) and the ability of the biofilter to remove ammonia from closed systems was assessed. The biofilters used in this study were plant-based.

The plant-based biofilters used in this study are considered to be hybridizations of phytoremediation (the biological degradation and accumulation of pollutants by plants) and traditional microbial biofiltration technologies. Mixed swards of living mosses that are commonly found in South Western Ontario were used as packing material for the biofilters tested. Mosses will bring a level of ecological balance to these complex biosystems since plants are well known to symbiotically enhance microbial populations. Also, mosses have a high surface area (approximately $1.6\text{m}^2/\text{g}$) (Llewellyn, 2000) that can act as a packing material for microbial growth (Llewellyn, 2000).

Nitrogen is an essential nutrient that is required by plants in large amounts and is usually the limiting nutrient in unfertilized plant systems. Nitrogen is a key building block for amino acids, proteins, and enzymes and is also an integral part of chlorophyll structure. Plants, in general, can directly absorb nitrogen as NH_3 by leaves; however most nitrogen is absorbed as inorganic NH_4^+ (ammonium) and NO_3^- (nitrate) through roots. NH_4^+ uptake is favoured by neutral pH where as NO_3^- uptake is favoured by low pH. It is believed that another important role that mosses will play in this study is: Mosses will readily absorb and utilize ammonia.

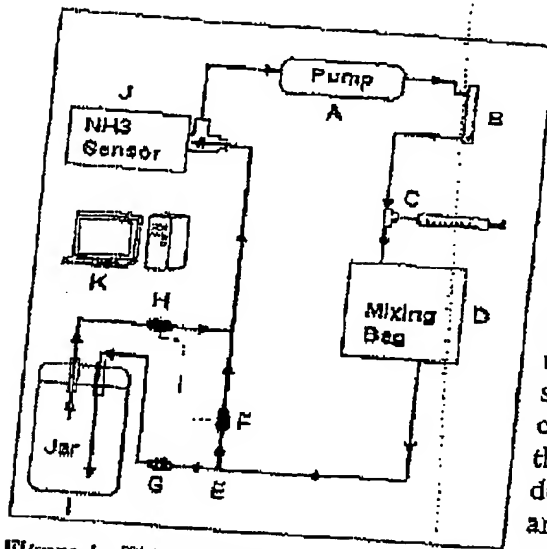
Materials and Methods

Figure 1 is a schematic of the experimental apparatus. Briefly, a SKC personal air-sampling pump (A) circulated the air within the system. The airflow was regulated at a constant flow rate ($0.01\text{m}^3/\text{minute}$) using an air flow meter (B). NH_3 (c.a. 50ppm v/v) was delivered into the system using a glass syringe and the injection port (C). A 10 L non-reactive air-sampling bag (D) was filled with 8 L of ambient air from the room to allow for adequate mixing of the ammonia with the air contained in the system.

A combination of a brass Swagelok tee (E), 1 normally closed brass one-way solenoid valve (F), and 2 normally open brass one-way solenoid valves (G and H) allowed for the airflow within the system to be directed through the biofilter or to bypass the biofilter prior to being sampled.

This arrangement of solenoids allowed for the measurement of influent and effluent ammonia concentrations of the air stream respectively with relative to the biofilters. The biofilters tested were contained in a 1 L glass vessel (I) that was placed inline with the 2 normally open one-way valves. The concentration of ammonia in the air stream was monitored using a P2065- NH_3 ammonia solid-state sensor (J) (Conspect, Toronto, ON). Triggering of the valves, data collection and experimental timing was done with the use of a personal computer (K) using Labview Development System Version 5.1 for Windows 98 (National Instruments, Austin, Texas). All the lines used in this system were 1/8"

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copper tubing and all the connections and tees were brass Swagelok fittings (Niagara Valve and Fitting LTD., Hamilton, Ontario). The concentrations of NH_3 in the effluent air stream from the biofilter was measured once every minute for 20 minutes. After 20 minutes, the valves were triggered to allow the air stream to bypass the biofilter to allow the concentrations of NH_3 of the influent air stream every minute for 10 minutes. This cycle of measurements were carried out until the concentrations of NH_3 fell below the detection limit of the sensor (0.5 ppm). Wet and dry weights of each experimental unit was measured, in order to determine moisture content and biomass for any given treatment.

Results

Influent and effluent concentrations of ammonia were measured to calculate removal efficiency for the biofilters over a measured period of time (Figure 2). Removal efficiency (%) was calculated as $(1 - \frac{\text{effluent concentration of ammonia}}{\text{influent concentration of ammonia}}) \times 100$ (Llewellyn, 2000).

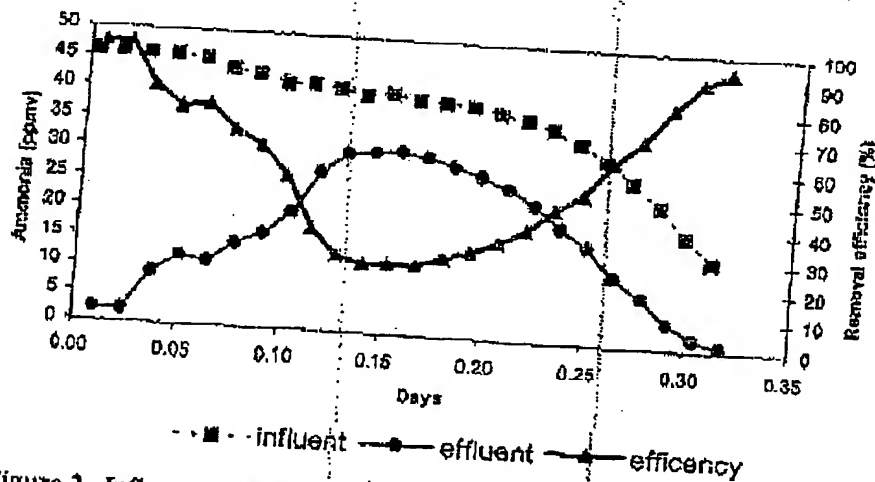


Figure 2. Influent and effluent concentrations of ammonia passing through a moss based biofilter. Removal efficiency is near 100% upon biofilter start up due to adsorption.

Figure 2 shows that ammonia concentrations in the effluent airstream of the biofilter were near zero for the initial time period of the runs. After 0.01 days, ammonia was detected in the effluent air stream and eventually reached a peak concentration of 30 ppmv at

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0.157 days where the removal efficiency was 24.2%. This concentration peak was then followed by a reduction of ammonia that eventually fell below the detection limit of the ammonia sensor (0.50 ppmv) resulting in a maximum removal efficiency of 94.5%. This trend was seen for all three replicates. We speculate that in the time period directly following exposure of the biofilter to ammonia, the ammonia in the air stream of the influent air is absorbed to the mosses and the moisture that surrounds the mosses until a point of saturation is reached. This adsorption and saturation can be used to explain the near 100% removal efficiency that is first observed. Once saturation is obtained, the removal efficiency decreases until a point of equilibrium is reached between adsorption and desorption of ammonia and the mosses. Once this equilibrium is reached, changes in the effluent air stream is due to removal of ammonia from the air stream by the biofilter, either by microbial degradation, plant uptake or a combination of both.

Ammonia effluent concentrations (after saturation has occurred) were plotted against corresponding ammonia influent concentrations (Figure 3), resulting in an exponential relationship, $r^2=0.9993$. This deviates from the linear relationship that was expected. Due to the equilibrium of gaseous ammonia with aqueous ammonium, it is suspected that as the system removes NH_3 from the influent air, an amount of NH_4^+ will come out of solution releasing a small amount of NH_3 . This will result in higher ammonia effluent concentrations causing the deviation from linearity.

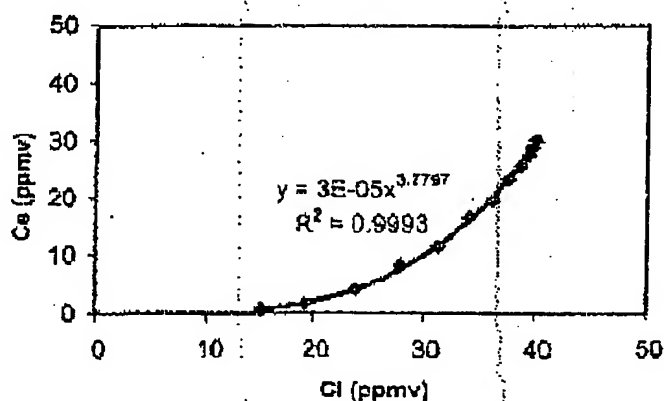


Figure 3. Ammonia Influent (C_i) concentrations are shown with corresponding ammonia effluent concentrations (C_e) for a moss-based biofilter. Data shown for both influent and effluent concentrations are taken after the biofilter is saturated with ammonia and an equilibration is achieved.

Elimination capacity, the quantity of contaminate degraded per unit biofilter over a period of time, was also calculated for each biofilter when the influent concentration was 25ppmv (Table 1).

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Table 1. Elimination capacities and removal efficiencies were calculated for the period at which influent ammonia concentrations were 25 ppmv for three moss-based biofilters.

System	Elimination Capacity $\text{g ammonia g}^{-1} \text{ moss h}^{-1}$	Removal efficiency (%)
Biofilter 1	1.57×10^{-5}	62.44
Biofilter 2	2.03×10^{-5}	44.80
Biofilter 3	1.41×10^{-5}	58.93

Conclusions

The results of this study show that ammonia at concentrations nearing 50 ppmv has been successfully removed from a closed system by moss-based biofilters. Once saturation of the biomass with ammonia is achieved, removal efficiencies of the biofilters increased until approximately 100% removal was observed. Instantaneous elimination capacities (when influent ammonia concentrations were 25.0 ppmv) were determined to be 1.57×10^{-5} , 2.03×10^{-5} , and 1.41×10^{-5} grams of ammonia per grams of moss per hour for three moss based biofilters.

Acknowledgements

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